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# Scanning Electron Microscopy (SEM) Procedure for HE Powders

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# **Scanning Electron Microscopy (SEM) Procedure for HE Powders on a LEO 438VP System**

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# Scanning Electron Microscopy (SEM) Procedure for HE Powders on a LEO 438VP System

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## **1.0 SCOPE & APPLICATION**

This method describes the characterization of HE powders by Scanning Electron Microscopy (SEM).

## **2.0 SUMMARY OF METHOD**

HE particles are dispersed onto an aluminum standard SEM specimen mount. Electron micrographs are collected at various magnifications (150 to 10,000 X) depending on HE particle size.

## **3.0 APPLICABLE SPECIFICATIONS, OTHER PUBLICATIONS**

### **3.1 Other Publications:**

- 3.1.1 Ted Pella Cressington Magnitron Sputter Coater, model 108 operator's guide
- 3.1.2 LEO 438VP operator's manual

## **4.0 APPARATUS AND MATERIALS**

The following equipment or equivalent equipment is required to analyze samples in accordance with this method:

- 4.1 SEM specimen mounts: 1/2 inch diameter x 1/2 inch tall, aluminum, P/N 16202 (Ted Pella)
- 4.2 Ted Pella Cressington Magnitron Sputter Coater (model 108) with gold Source.
- 4.3 LEO 438 VP (variable pressure) environmental scanning electron microscope
- 4.4 Ted Pella Specimen Box, Figure 1
- 4.5 MRS-3XY grid, magnification standard from Geller MicroAnalytical

## **5.0 REAGENTS**

### **5.1 Hexane**

## **6.0 SAMPLE DISTRIBUTION & HANDLING**

Samples are distributed and the handling and transportation will be in accordance to the LLNL explosives safety procedures.

## **7.0 PROCEDURE**

### **7.1 HE Sample Preparation**

- 7.1.1 Obtain SEM HE Logbook, B191/R1500. Record sample(ID) numbers in logbook.
- 7.1.2 Disperse a small quantity of HE particles, <5 milligrams, on a standard 1/2 inch diameter by 1/2 inch tall aluminum SEM specimen mount. Repeat for each sample to be imaged
- 7.1.3 Place the samples in the gold sputter coater (Ted Pella Cressington Magnitron sputter coater) and evacuate to 0.01 mbar. Once the desired vacuum is achieved, turn on the argon gas. Flush the coating chamber a few times with argon and then evacuate it. The distance from the sample platform to the gold source is about 7.5 cm.

- 7.1.4 Finally, put enough argon in the chamber so that the vacuum reads 0.08 mbar. Coat the samples with gold for 30 seconds (pausing at 15 seconds for 1 minute) at 20 mA. Recoat as necessary to achieve a good image after analyzing on the SEM. This batch of HE samples were coated in three layers.
- 7.1.5 Transfer samples back to appropriate positions in the specimen box.

## **7.2 SEM Characterization**

- 7.2.1 Vent the SEM (LEO 438 VP) and place the MRS calibration grid on the SEM sample carousel and evacuate the SEM.
- 7.2.2 Perform a beam alignment on the SEM for optimum brightness. Refer to the SEM manual for detailed procedures on beam alignment.
- 7.2.3 Determine the beam's first, and second, saturation points. Keep in mind that this slightly changes over time and needs to be checked and adjusted accordingly before each use. It is recommended to analyze HE samples on the first saturation point due to its sensitivity.
- 7.2.4 Using the MRS-3XY grid, magnification standard from Geller MicroAnalytical measure the calibration gridlines before and after the samples. These measurements are used to calculate the correction factor. Record the calibration correction factor in the logbook at the beginning and end of time period over which the samples are analyzed, Figure 3.
- 7.2.5 Once all the operating parameters are set, save it, then vent the SEM chamber and remove the grid.
- 7.2.6 Orientation in SEM carousel is as follows: Positions 1 through 4 are clockwise as indicated in Figure 4. Transfer samples from specimen box to SEM carousel positions. Record SEM carousel position and the corresponding HE serial number in the HE SEM log book (001447).
- 7.2.7 Gold coated samples are examined in a LEO 438 VP environmental scanning electron microscope. High-resolution imaging is performed with accelerating voltages (2-5 Kv), appropriate probe current, and various working distances (10-14mm) and tilt angles as appropriate for satisfactory electron micrographs (standard operating tilt angle is 0). One may change these values to achieve better resolution and high quality image and minimize damage to the sample.
- 7.2.8 Electron micrographs will be taken at magnifications 150X, 300X, 600X, 1,000X, and 3,000X. An image will also be taken at 10,000X if possible. Care shall be taken to assure that the electron beam does not damage HE. Slowly adjust voltage, current and working distance to minimize charging and damage to the particles.

- 7.2.9 General morphology of HE powder shall be noted while examining several areas of the samples. If particle dispersion is uniform, examine several random areas. Measure typical “largest” and “smallest” particle sizes. Take electron micrographs of at least two areas exhibiting a good sampling of particles, at appropriate magnifications showing numerous particles to individual particles. Complete narrative of HE particle morphology including notations of any anomalies for each sample should be noted.
- 7.2.10 Note the corresponding HE parent serial number on each electron micrograph.

### **7.3 Sample Archival**

- 7.3.1 Return any excess HE to the Sample Custodian.

### **7.4 Data Recording and Storage**

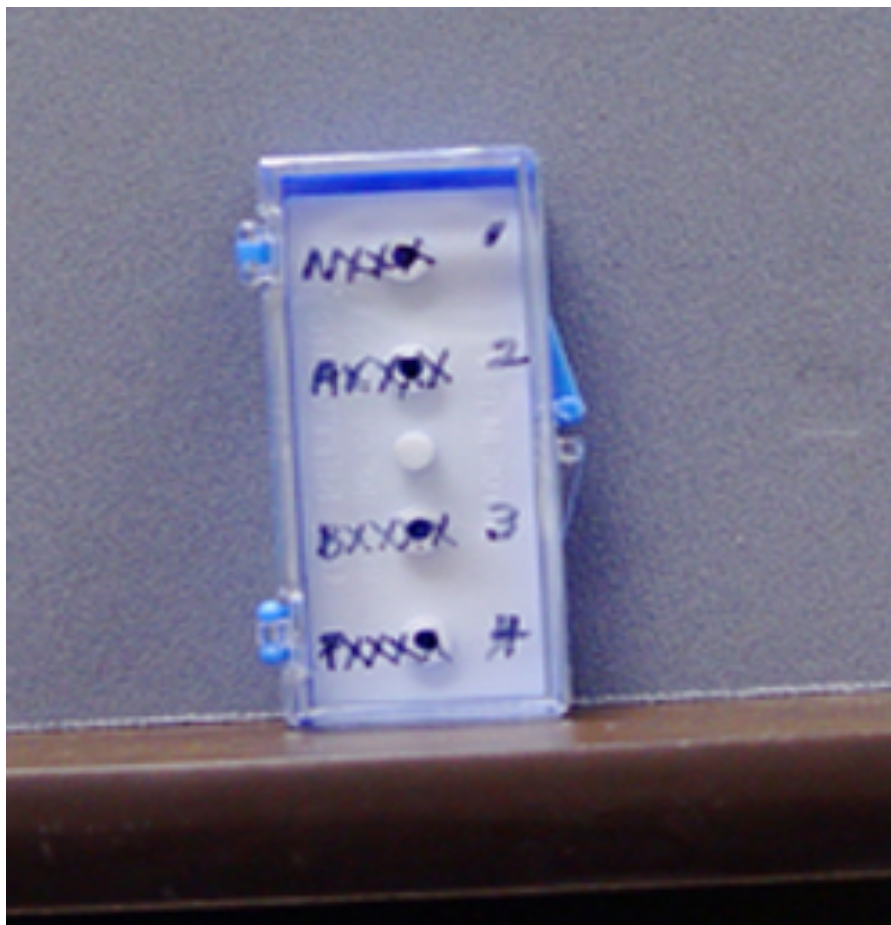
- 7.4.1 Sign off Traveler for the samples.
- 7.4.2 Record process completion date, operator’s name and procedure version in logbook.
- 7.4.3 A description of each electron micrograph including its laboratory ID (HE parent detonator numeric serial number) will be recorded in the “HE SEM Logbook”. Example: XXXXX-1, XXXXX-2, XXXXX-3, etc. Each image will be stored using SEM software on the computer associated with the SEM. Record all images on CD-R. Provide two hard copies of all data with a copy of corresponding logbook pages to surveillance engineer or designee if requested. One hard copy, logbook, and CD will be stored in B191, Rm. 1443 and other will be stored in a separate location.

## **8.0 QUALITY CONTROL AND METHOD PERFORMANCE**

The quality of measurements or qualitative judgments made from SEM electron micrographs is subject to operator training and expertise.

## **9.0 REFERENCES**

- 9.1 Test Plan and Procedure for Distribution and disassembly of HE Powder Aliquots.

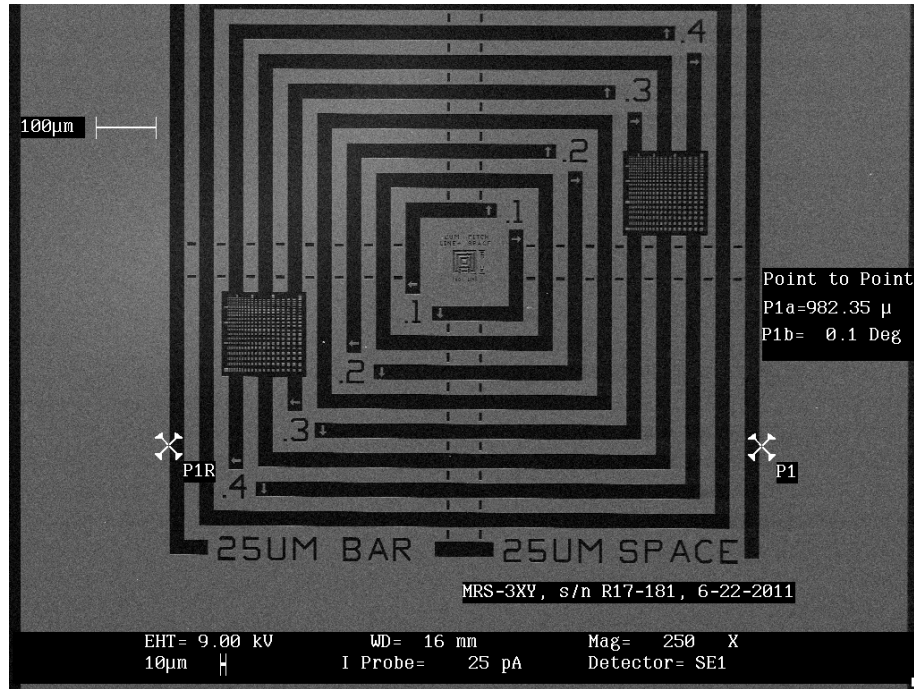


**Figure 1. Specimen Box**

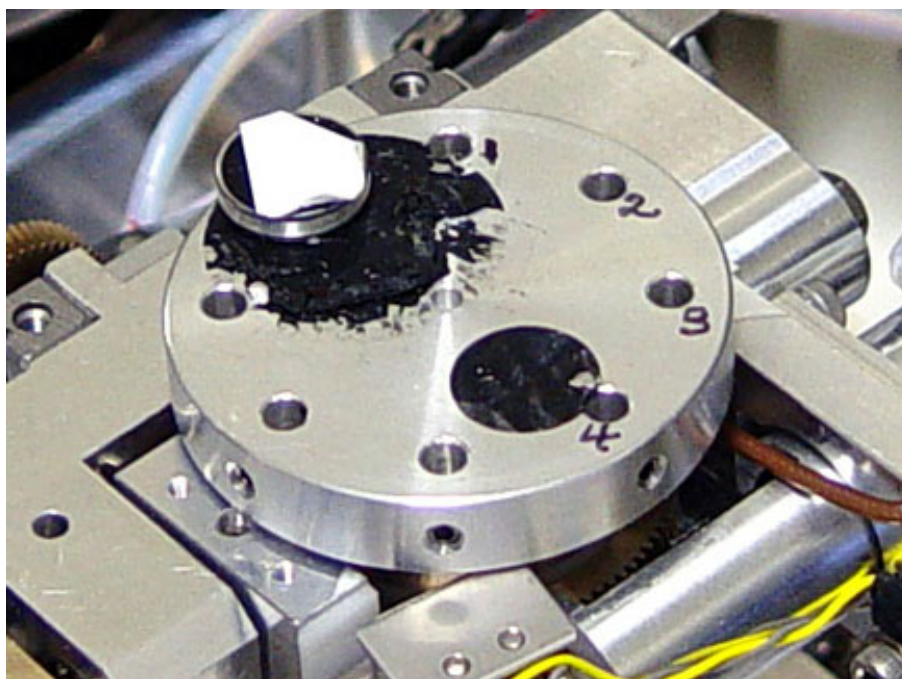




**Figure 2. Sputter Coater Positions**



**Figure 3. MRS-3XY grid image**



**Figure 4. SEM Carousel Positions**